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Covering the period February 1, 1977 to April 30, 1977

NOVEL DUPLEX VAPOR-ELECTROCHEMICAL METHOD FOR SILICON SOLAR CELLS

By: Vijay K. Kapur, L. Nanis, and Angel Sanjurjo

Prepared for:

JET PROPULSION LABORATORY
California Institute of Technology
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Pasadena, CA 91103

Attn: Dr. Ralph Lutwack
Spacecraft Power Station

Contract No. 954471 under NAS 7-100
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ACKNOWLEDGEMENTS

The assistance of Drs. D. Hildenbrand, K. Lau and P. Kleinschmidt for obtaining the data on the thermal decomposition of Na_2SiF_6 and for the impurity analysis of SiF_4 gas by mass spectrometry is gratefully acknowledged.

SUMMARY

Silicon was produced by alternate pulse feeding of the reactants SiF_4 gas and liquid sodium. The average temperature in the reactor could be controlled to within ± 20°C in the temperature range 400-600°C, by regulating the amount of reactant in each pulse. Batches of reaction product (NaF, Na_2SiF_6, Si) as large as 250 g, containing 25 to 30 g of silicon, were prepared by the alternate pulse feeding technique.

Silicon tetrafluoride gas was analyzed by mass spectrometry to determine the nature and amount of contained volatile impurities. The major impurities were found to be silicon oxyfluorides (≃ 4.0%), sulfur oxyfluorides (≃ 0.1%), and sulfur dioxide (0.05%). Sodium metal was analyzed by emission spectrography, and it was found to contain only calcium (100 ppm wt) and copper (8 ppm wt) as impurities.

The decomposition of Na_2SiF_6 to NaF and SiF_4 was studies by an effusion method to determine the equilibrium pressure of SiF_4 at temperatures ranging from 585 to 650 K (312 to 377°C).

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INTRODUCTION

At SRI International, silicon has been produced by the reduction of SiF_4 gas with liquid sodium[1,2] according to the reaction

$$SiF_4(g) + 4\ Na(l) \rightarrow Si(c) + 4\ NaF(c) \qquad (1)$$

In a side reaction, sodium fluosilicate (Na_2SiF_6) is also produced[3] from the reaction of NaF with SiF_4 according to

$$2\ NaF(c) + SiF_4(g) \rightarrow Na_2SiF_6(c) \qquad (2)$$

The reaction products, therefore, consist of Si, NaF and Na_2SiF_6. The SiF_4 - Na reaction (Equation 1) is performed in a reaction kettle[4] by providing an atmosphere of SiF_4 gas over the surface of liquid sodium. The reaction kettle is heated to about 200°C in order to initiate the reaction shown in Equation 1. Once the reaction has started, a large amount of heat is liberated, which speeds the reaction to completion. However, in a simplified consideration, as the reaction proceeds, the solid reaction products Si, NaF and Na_2SiF_6 cover the surface of the liquid sodium, thus decreasing the accessibility of SiF_4 gas. For the reaction to continue further, SiF_4 gas has to diffuse through an increasingly thick layer of reaction products to reach clean sodium. It is postulated that, after a while, the surface of the liquid sodium becomes so heavily coated with reaction products that a diffusion limitation of the reaction is established, which is controlled by the thickness and the porosity of the product layer, as well as by temperature and pressure of SiF_4. As a consequence, some unreacted sodium is

left buried in the reaction products. The presence of sodium metal in the reaction products is undesirable because it reacts vigorously with HCl solutions used for leaching out the sodium fluorides. Of course, the acid leachout is itself desirable, because, with water as leachant, alkaline solution is formed, which causes the unwanted dissolution of silicon. To minimize the amount of unreacted sodium in the reaction products, it was decided to perform the SiF_4-Na reaction (Equation 1) by alternately pulse feeding the reactants in the sequence: liquid sodium pulse followed by SiF_4 gas pulse.

The pulse feeding technique used for the SiF_4-Na reaction is described in the following section of this report. To pinpoint possible sources of impurity pickup, we analyzed both the SiF_4 gas and the sodium metal used in the reaction (1). SiF_4 gas is known to contain oxygen and sulfur dioxide as impurities, but to check for possible contamination of volatile metal halides, SiF_4 gas was analyzed by mass spectrometry. Sodium metal also was analyzed by emission spectrography. The results of these analyses are discussed in this report.

Because of the importance of sodium fluosilicate to this project, both as a reaction product (Equation 1,2) and as a possible intermediate material for SiF_4 generation, a study was undertaken to determine the equilibrium partial pressure of $SiF_4(g)$ above $Na_2SiF_6(c)$ and its decomposition products $SiF_4(g)$ and NaF(c). Data available in the literature are widely different for the reaction which is the reverse of Equation 2, namely

$$Na_2SiF_6(c) \rightleftarrows SiF_4(g) + 2\ NaF(c) \qquad (3)$$

The compound Na_2SiF_6 has been detected as a product of the reaction between Na and SiF_4. Since silicon is the desired product, experiments

to minimize the formation of Na_2SiF_6 are in progress. It is expected that an increase in reaction temperature will hasten the decomposition of Na_2SiF_6. The alternate pulse feed method is being used to control reaction temperature. However, the lowest possible temperature is desirable in order to decrease impurity pickup from container materials.

Alternatively, a silicon purification scheme is also possible in which the product silicon is anodically dissolved in a fluoride melt and cathodically deposited to accomplish electrolytic refining, utilizing Na_2SiF_6 and NaF to form a suitable molten electrolyte. In this case, conditions for the reactor operation might be controlled so as to encourage the formation of Na_2SiF_6 during the reduction of SiF_4 by Na.

The decomposition of Na_2SiF_6 takes on added significance as a source of SiF_4 for reaction with Na, because inexpensive sources of silicon (H_2SiF_6) may be used to form Na_2SiF_6. Members of the Thermochemistry Group in our Materials Research Center (D. Hildenbrand, K. Lau) performed the effusion vapor pressure measurements presented in this report, which establish the temperature-pressure relationship for SiF_4 for the reaction in Equation 3.

SODIUM PULSE FEEDER

To introduce pulses of liquid sodium into the reaction kettle, we constructed a sodium pulse feeder (Fig. 1) from a cylindrical stainless steel container, 5½ inches long, 3 inches in diameter, with ½ inch thick walls. The top of the feeder is closed by bolting on a stainless steel lid with an O-ring seal. A thermocouple (TC1), inserted through the lid, is used to measure the temperature of liquid sodium. The sodium feeder can be evacuated through a needle valve (V_1) and it can also be filled with an inert gas through another needle valve (V_2). The pressure gauge (PG) is used to measure the back-up pressure of the inert gas in the feeder. The inside floor of the feeder is conically tapered to assist the flow of liquid sodium. A hole in the center of the floor connects to a welded stainless steel tube (3/8 in O.D.), through which liquid sodium is drawn out. The capacity of the sodium feeder is about 300 ml and it can easily contain 250 g of sodium. The outflow of liquid sodium can be regulated by a stainless steel bellows valve V_3.

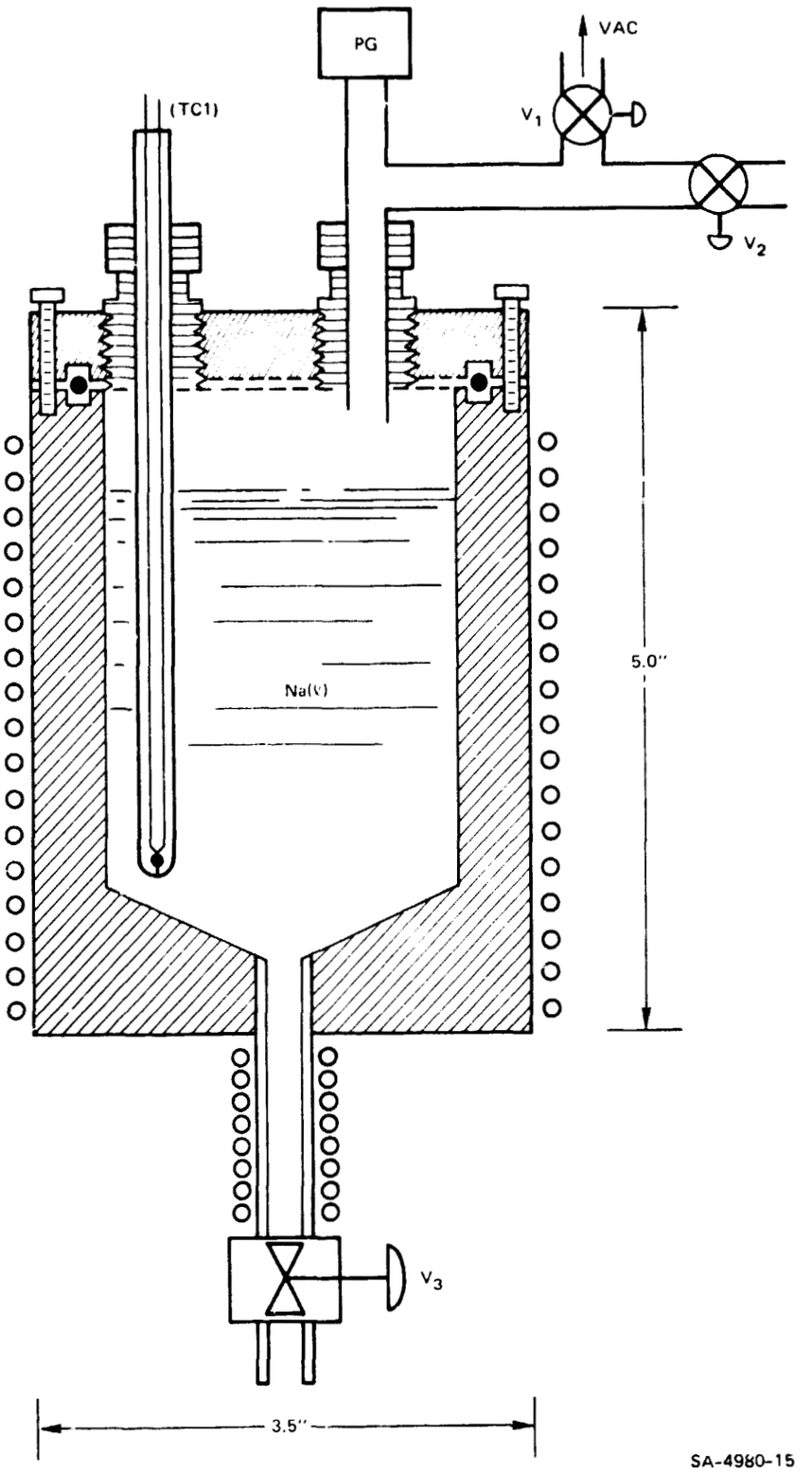

FIGURE 1 SODIUM FEEDER

PULSE FEEDING OF REACTANTS

To perform the SiF_4-Na reaction by pulse feeding the reactants, we connected the reaction kettle (described in Quarterly Progress Report 2 and 3) to the sodium pulse feeder containing liquid sodium and also to the SiF_4 storage through a pre-reservoir of SiF_4 gas, as shown in Fig. 2. The reaction kettle (c) was first evacuated via a needle valve, V_6. To perform the SiF_4-Na reaction (Equation 1), we introduced pulses of liquid sodium and SiF_4 gas by opening valves V_3 and V_4 as required. During the reaction, the pressure of SiF_4 gas in the system, measured by the pressure transducer (PT), and the average reaction temperature, measured by the thermocouple (TC2), were continuously recorded.

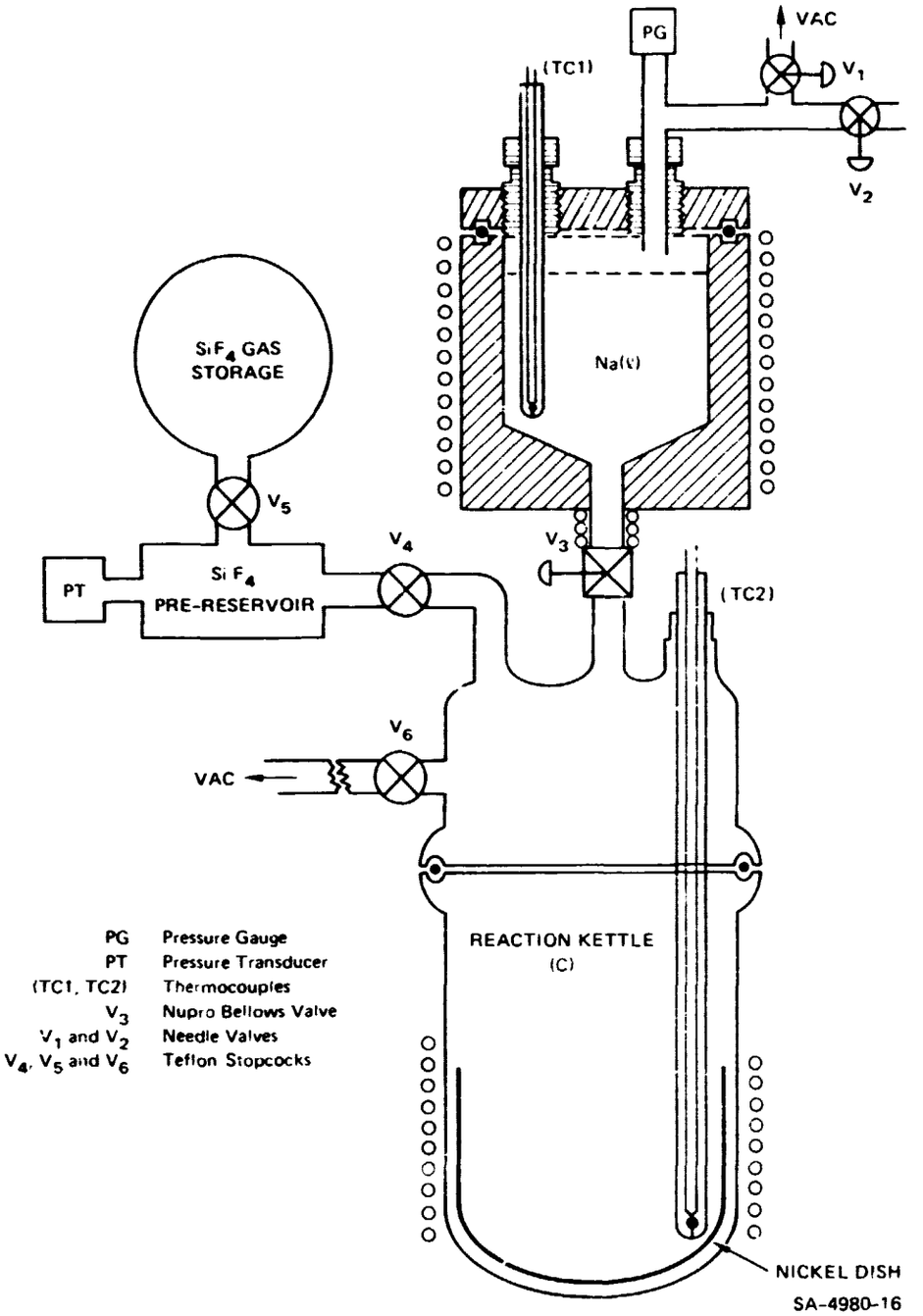

FIGURE 2 APPARATUS FOR SiF_4 — Na REACTION

PULSE FEEDING OF SiF_4 GAS ONLY (MONOPULSING)

In our first attempt to use the pulse feeding technique, only SiF_4 gas was pulse fed over a pool of liquid sodium. To start the experiment, we first evacuated the reaction kettle (C, Fig. 2) and introduced liquid sodium (12.08 g) from the sodium feeder by opening the bellows valve, V_3 (Fig. 2). As shown in the temperature trace of Fig. 3, sodium was further heated at 305°C (a temperature well above the initiation temperature of the Na-SiF_4 reaction) before pulses of SiF_4 gas were fed into the reaction kettle. SiF_4 gas pulses were introduced by opening stopcock V_4 (Fig. 2) connecting the reaction kettle to the SiF_4 gas pre-reservoir. Pressure in the system was measured by a pressure transducer (PT) connected to the SiF_4 gas pre-reservoir. Traces of the pressure and temperature of a typical monopulse experiment are shown in Fig. 3. The trace of SiF_4 pressure recorded againt time can be divided into four time intervals, as shown schematically in Fig. 4.

During the time interval t_1, the SiF_4 pre-reservoir was filled with SiF_4 gas and was kept isolated from the reaction kettle containing liquid sodium. The pressure transducer showed the pressure in the pre-reservoir increasing to P_1. Towards the end of the holding period, t_2, the stopcock V_4 (Fig. 2) was opened and a pulse of SiF_4 gas was introduced into the reaction kettle. Because of the larger volume of lines and container now available to SiF_4 gas, its pressure suddenly dropped from P_1 to P_2 during the brief time interval t_3. Since the sodium metal was already at 305°C, the SiF_4-Na reaction started instantaneously and consumed SiF_4 gas, causing a further slower drop of P_{SiF_4} from P_2 to vacuum. During the time interval t_4, P_{SiF_4} decreased as

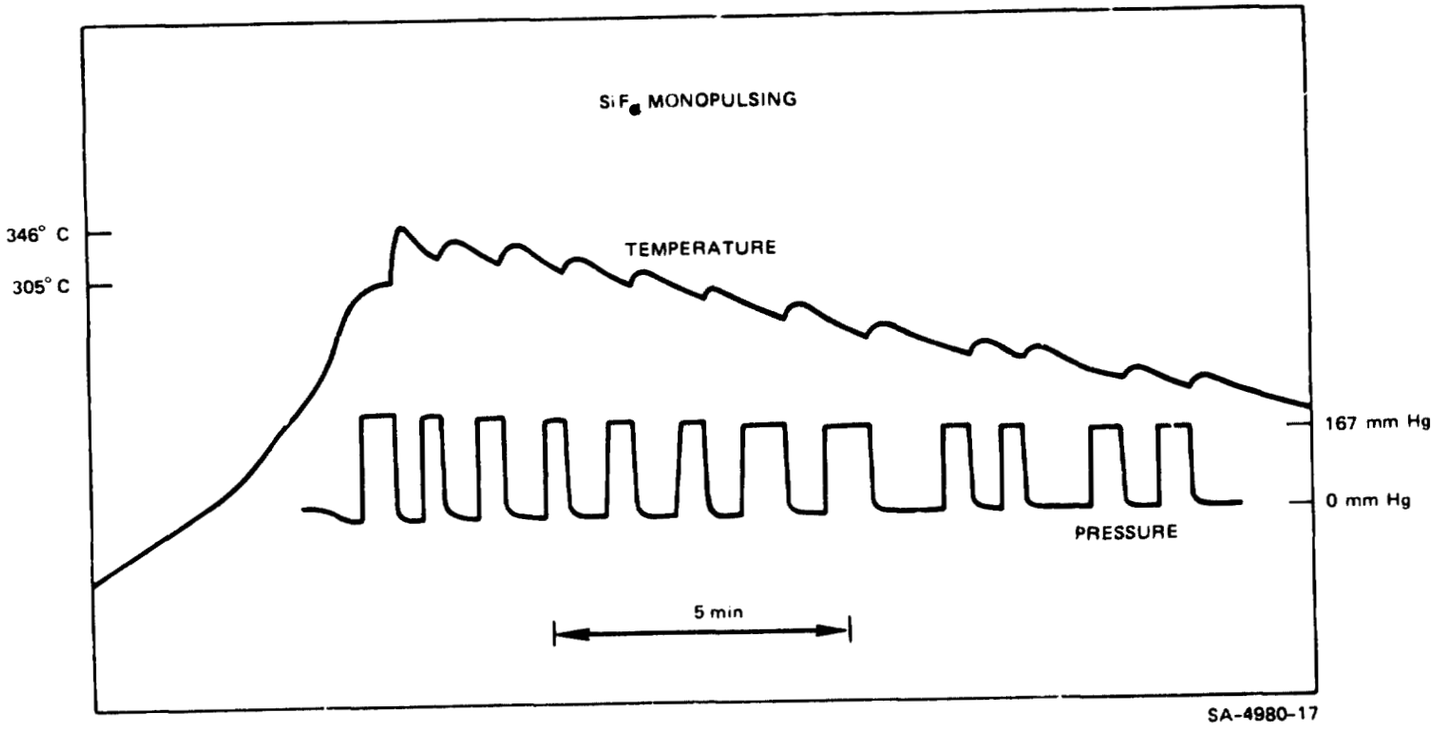

FIGURE 3 SiF_4 PRESSURE AND REACTION TEMPERATURE VERSUS TIME (MONOPULSING)

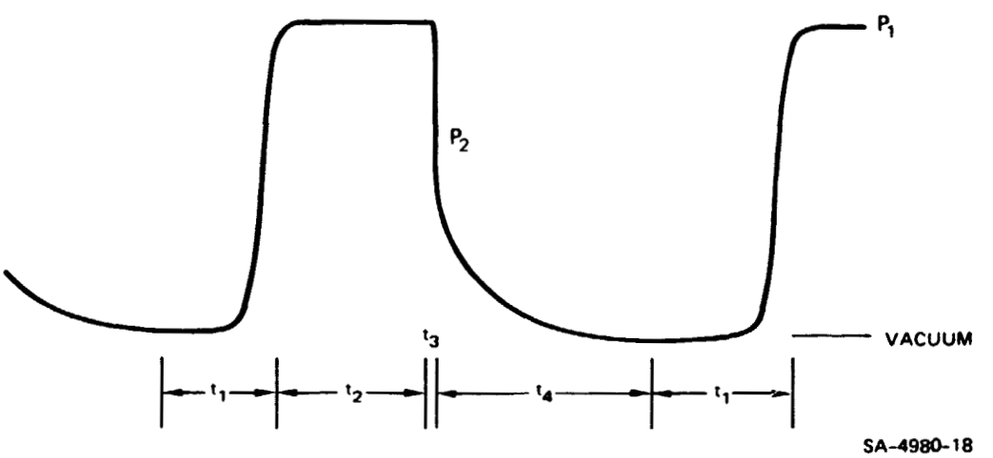

FIGURE 4 MONOPULSING SiF_4 PRESSURE VERSUS TIME (SCHEMATIC)

controlled by the kinetics of the reaction. As each pulse of SiF_4 gas reacted with Na, the heat of the reaction produced a corresponding increase in the reaction temperature,as observed in the temperature trace shown in Fig. 3. Following reaction, the kettle was again disconnected from the SiF_4 pre-reservoir by closing the stopcock V_4, thus again commencing the cycle shown in Fig. 4. After a few pulses of SiF_4 had been fed, the reaction products covered the surface of the liquid sodium. When the introduction of an SiF_4 pulse produced no corresponding increase in reaction temperature, it was assumed that all of the available sodium had reacted.

Substantial amounts of unreacted sodium (greater than 20%) were found in the reaction products of monopulsing experiments. Therefore, we adopted a procedure for pulse feeding both SiF_4 gas and liquid sodium (bipulsing). The sodium feeder described above (Fig. 1) was used in bipulsing experiments.

PULSE FEEDING OF LIQUID SODIUM AND SiF_4 GAS (bipulsing)

Liquid sodium and SiF_4 gas were both pulse fed into the reaction kettle to avoid carrying over unreacted sodium in the reaction products. Typical pressure and temperature recordings are shown in Fig. 5. Every pulse of sodium added a fresh layer of sodium on top of the existing reaction products so that the following pulse of incoming SiF_4 gas directly contacted liquid sodium. The average amount of liquid sodium in each pulse was 3 g and each sodium pulse was followed by 8 to 10 pulses of SiF_4 gas to produce complete reaction. The SiF_4 pulses were fed directly into the reaction kettle, without going through the intermediate step of filling the pre-reservoir (as in monopulsing experiments). During the course of reaction following a pulse, the pressure of SiF_4 in the reaction kettle decreased from a maximum of 580 mm Hg to vacuum. For each pulse of SiF_4, a rapid pressure increase was followed by the exponential decay of P_{SiF_4} due to the SiF_4-Na reaction, as shown in enlarged detail in Fig. 6. An average temperature of 600°C was maintained for the reaction whose history is partly shown in Fig. 5.

An extremely useful feature of the bipulsing technique is that the average temperature inside the reactor can be maintained nearly constant by regulating the amounts of sodium and SiF_4 in each pulse. Since the temperature was held constant by reaction control, it could not serve as an indicator of the complete consumption of sodium in the SiF_4-Na reaction, as was possible in the monopulsing mode. It was assumed that about 8 to 10 pulses of SiF_4 were enough to completely react with an average sodium pulse amount of 3 g. After feeding about 10 pulses of SiF_4 gas, we fed another pulse of liquid sodium (Fig. 5) and then repeated the SiF_4 pulsing process. Batches of reaction products weighing 250 g,

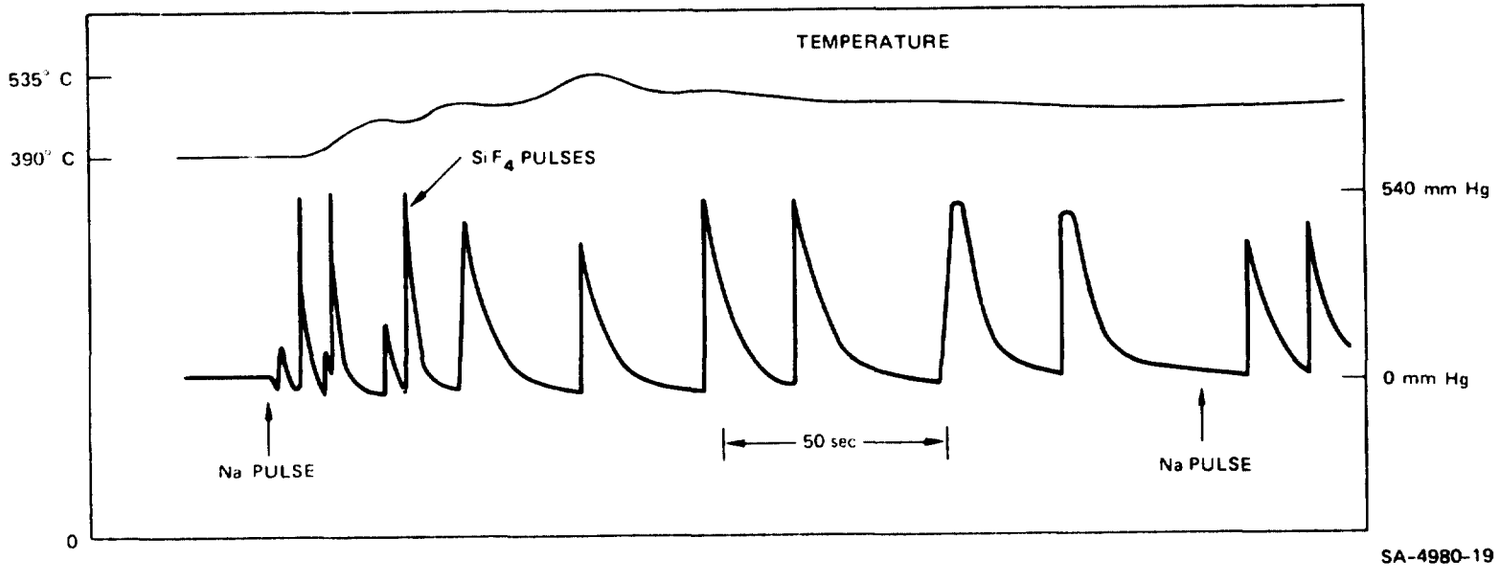

FIGURE 5 SiF_4 PRESSURE AND REACTION TEMPERATURE VERSUS TIME (BIPULSING)

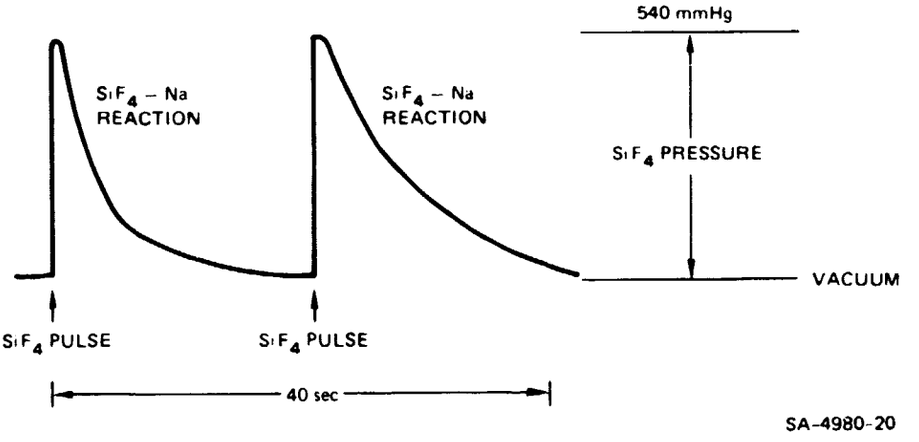

FIGURE 6 BIPULSING – SiF_4 PRESSURE VERSUS TIME (SCHEMATIC)

and containing 25 to 30 g of silicon, were produced. The amount of unreacted sodium in the reaction products was negligibly low for the bipulsing mode. A present limitation is the size of the reaction kettle; however, it should be possible to produce batches containing 100 to 150 g of silicon by scaling up to a larger size kettle.

ANALYSIS OF SILICON TETRAFLUORIDE BY MASS SPECTROMETRY*

As a convenient method of rapidly surveying the impurity content in the SiF_4 used in reaction 1, mass spectrometry was used. Two different samples were analyzed. Sample No. 1 was taken directly from the cylinder supplied by Linde Division, Union Carbide. Sample No. 2 was obtained from the pyrex storage bulb of the reaction system used for the Na-SiF_4 reaction. From the mass to charge ratio, isotopic distribution, gas beam shutterability, and ionization potential, various mass peaks were assigned to neutral precursors. From the peak intensities, the relative abundance of the various volatile compounds were calculated.

Experimental

The samples were analyzed by a Nuclide 12-60-HT (12" radius, 60° sector, high temperature source) mass spectrometer. With a 4500-volt ion accelerating potential, ions with a mass-to-charge ratio of up to 1000 can be detected. Ions were produced by electron bombardment in a Nier-Inghram ion source. The ion signal was amplified by an electron multiplier and detected with either a vibrating reed electrometer (VRE), or by counting ion pulses with an Ortec 9315 counter. With this detection system, ion intensities as low as 0.001% of the major peak are normally measured.

The sample gas in a stainless steel reservoir at 20.0 ± 0.3 mm Hg pressure was introduced into the ion source through an orifice (0.013 in. diameter) adjacent to the inlet of the ion source. At the inlet was a

*The work reported in this section was performed by P. Kleinschmidt and D. Hildenbrand.

movable slit which allows one to determine that portion of the signal coming directly from the orifice and that portion due to background gases.

The procedure used for analyzing the sample was to monitor the SiF_3^+ (major ion of SiF_4) signal and adjust the flow rate until the ion intensity was near the maximum level (that is, at molecular flow conditions and without damaging the detection system). The magnetic field was scanned from a mass to charge ratio of 19 to 370. The ionizing electron energy was set to 20 eV to reduce the extensive fragmentation of SiF_4 occurring at higher energies. This mass spectrum was then compared with a background mass spectrum. Those masses present in the sample but not in the background were then examined to determine if the signal was shutterable. Also, the flow rate could be regulated with the metering valve. If the signal showed a response to this change in flow rate, we concluded that the signal was caused by molecules in the sample.

Results

Ions formed from the sample gas were identified from the observed mass numbers, the isotopic distribution, and the threshold appearance potentials. Gaseous impurities positively identified in the sample were SO_2, $SiOF_2$, SO_2F_2, CCl_4, $Si_2O_2F_4$ and Si_2OF_6.

The pressure ratios of all molecules relative to SiF_4 were calculated from the peak heights of the ion in the mass spectrum. The intensity was corrected for the isotopic distribution and for fragmentation to give the total ion yield of a given molecule at 20 eV. Further corrections were made to the ion intensity to account for the difference in ionization cross sections between SiF_4 and the impure gases. This correction ranged between 0.7 to 1.9 and was calculated by adding atomic cross sections. The corrected intensities were added and the relative

pressures of the various gases were calculated (see Table 1). No significant difference was detected between the two samples studied.

Discussion and Conclusions

In this analysis, impurities with concentrations less than 0.02% were not examined. An unassigned ion was found at mass peak 49. This could be BF_2^+. The concentration of this molecule was less than 0.02%. Other unassigned peaks were found at masses 40 and 45.

The principal result of this analysis is that the silicon oxyfluorides are major impurities in commercial SiF_4. No evidence was found of phosphorus, titanium, zirconium, vanadium, iron or chromium impurities in concentrations greater than 0.02%. The extensive fragmentation and isotopic spectra of the major impurities, of course, overlapped a substantial portion of the mass spectrum, so that minor impurities at these mass numbers could not be detected. Although volatile impurities of elements such as Ti, V, Zr, Fe, Cr, Al and P in amounts greater than 200 ppm (0.02%) were not detected, this analysis does not preclude their presence in SiF_4 gas. Methods for detection of low levels of these impurities and their removal from SiF_4 gas will be investigated.

Table 1

MASS SPECTROMETRIC ANALYSIS OF COMMERCIAL SiF_4

Ion	Mass Number	Intensity (counts per sec)		Neutral Precursor	Relative Abundance pct
		As Recorded	Corrected		
SO_2^+	64	20	14	SO_2	0.035
$SiOF_2^+$	82	37	709	$SiOF_2$	1.79
$SO_2F_2^+$	102	39	39	SO_2F_2	0.098
CCl_3^+	118	16	63	CCl_4	0.159
$Si_2O_2F_4^+$	164	15	32	$Si_2O_2F_4$	0.081
$Si_2OF_6^+$	186	500	1681	Si_2OF_6	4.24
SiF_3^+	85	33000	37066	SiF_4	93.6

ANALYSIS OF SODIUM METAL BY EMISSION SPECTROGRAPHY

Sodium metal used in the SiF_4-Na reaction was analyzed by emission spectrography. The only impurities that could be detected were calcium (100 ppm wt), the most commonly found impurity in sodium, and copper (8 ppm wt). However, the analysis indicated that the sodium was quite pure, and it did not contain undesirable impurities such as Ti, V, Zr, Fe, Cr, Mn.

DISSOCIATION PRESSURE AND THERMODYNAMIC STABILITY OI $Na_2SiF_6(c)$*

The conditions of temperature and pressure under which $Na_2SiF_6(c)$ is thermodynamically stable are of interest in optimizing the yield of elemental silicon from the reaction of silicon tetrafluoride and sodium, and also for the generation of SiF_4 by thermal decomposition according to the reverse of the reaction previously indicated as Equation 2, that is,

$$Na_2SiF_6(c) = 2NaF(c) + SiF_4(g) \quad . \qquad (3)$$

Optimization of product yield can generally be predicted from standard thermodynamic calculations. However, in the Na-Si-F system, such calculations are precluded by the lack of adequate data on the standard enthalpy of formation, ΔH^o_{f298}, of $Na_2SiF_6(c)$. There have been several pertinent experimental studies on $Na_2SiF_6(c)$, including reaction calorimetry[5] and dissociation pressure measurements[6,7] but the results are rather discordant and of uncertain accuracy. The entropy of $Na_2SiF_6(c)$ is known from heat capacity data. From this information, an estimate of

$$\Delta H^o_{f298} \ (Na_2SiF_6,c) = -695.4 \text{ kcal/mol}$$

has been derived. Using this result and other available thermochemical data for NaF(c),[8] $SiF_4(g)$,[9] and $Na_2SiF_6(c)$,[10] one can calculate the equilibrium dissociation pressure for the reaction given in Equation 3 over a wide temperature range. The measured dissociation pressures

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*This reaction is based on work performed by D. L. Hildenbrand, K. H. Lau, and A. Sanjurjo.

scatter widely about the calculated pressures, with the differences amounting to as much as an order of magnitude or more.

Because of these substantial discrepancies, a new determination of the Na_2SiF_6 dissociation pressure was initiated at SRI by the torsion-effusion method.[11,12] To date, we have measured the dissociation pressure of commercially obtained* Na_2SiF_6(s) with two alumina effusion cells having 1.0 and 1.5 mm diameter orifices in the range 585 to 650 K. The results are shown in Figure 7. The slopes of the log P vs. 1/T plots with the two cells are in close agreement, but there is a definite trend such that higher absolute pressures are obtained with the 1.0 mm diameter orifice. This effect is quite common in solid decomposition reactions, and is taken to indicate the existence of a substantial kinetic barrier to the transfer of vaporizing species from a lattice site to the gas phase.[13] It is possible, however, to extrapolate the steady-state pressure data to the zero orifice area, as suggested by a theoretical model of the effusion process,[14,15] and to derive equilibrium pressure from the results. The extrapolated equilibrium pressure data are also shown in Figure 7.

From least squares fitting, the following expressions for the dissociation pressure were obtained:

1.0 mm diam. orifice: $\log P(atm) = (9.31 \pm 0.11) - (9180 \pm 70)/T$

1.5 mm diam. orifice: $\log P(atm) = (9.55 \pm 0.43) - (9440 \pm 27)/T$

The corresponding equation for the extrapolated equilibrium pressure is $\log P_{eq}(atm) = 9.11 - (9180/T)$.

It is assumed that the observed dissociation process is given by Equation 3, but this will be checked by determining the molecular weight

*99% Na_2SiF_6 obtained from Ventron Corporation, Masachusetts.

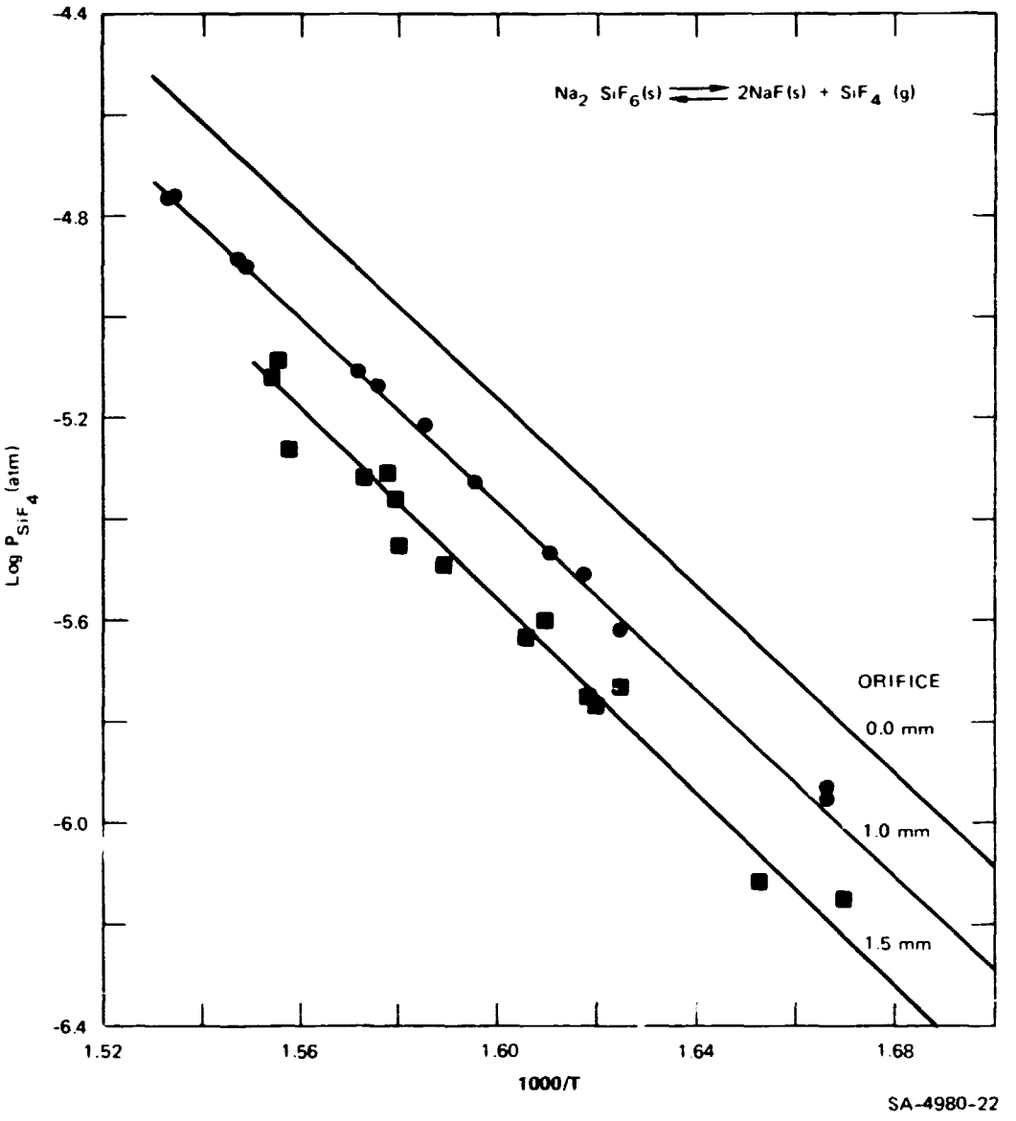

FIGURE 7 TORSION EFFUSION MEASUREMENTS OF P_{SiF_4}

of the effusing vapor from simultaneous torque-angle and weight-loss measurements.

Examination of the cell residues by x-ray diffraction showed the presence of Na_2SiF_6(s) and NaF(s) only, so that the decomposition process appears to be correct as written in Equation 3. In particular, there was no evidence of additional compounds such as ($NaF \cdot 4Na_2SiF_4$), the presence of which would substantially alter the interpretation.

The second law slopes of the dissociation pressure data (that is slopes of the plots of log P vs. 1/T yield values of 42.0 and 32.2 kcal/mol for the enthalpy change of the decomposition process at the average temperature of the measurements, using the 1.0 and 1.5 mm diam. orifices, respectively.

Data obtained with the smaller effusion orifice are believed to be the more reliable because of the higher precision. The slope heat of 42.0 kcal/mol can be combined with heat capacity data to yield ΔH^o_{298} = 44.0 kcal/mol for the decomposition reaction (Equation 3). A preliminary third law calculation, based on the absolute pressure and estimated entropy for Na_2SiF_6(c) yields ΔH_{298} = 40.4 kcal/mol. The ΔH^o_{298} values calculated by the second law and the third law differ by 3.6 kcal/mol . However, the effusion data show Na_2SiF_6(c) to have a significantly higher thermodynamic stability than indicated by previous data. (ΔH^o_{298} = 36.4 kcal/mol .)[5] Measured dissociation pressures are two orders of magnitude less than values calculated from NBS data.[8]

In Figure 8, the equilibrium pressures derived from the effusion studies are plotted along with the data of Hantke[6] and Caillat[7] obtained at higher temperatures. The literature data are quite scattered, but the extrapolation of SRI effusion data appears consistent with them at the highest temperatures.

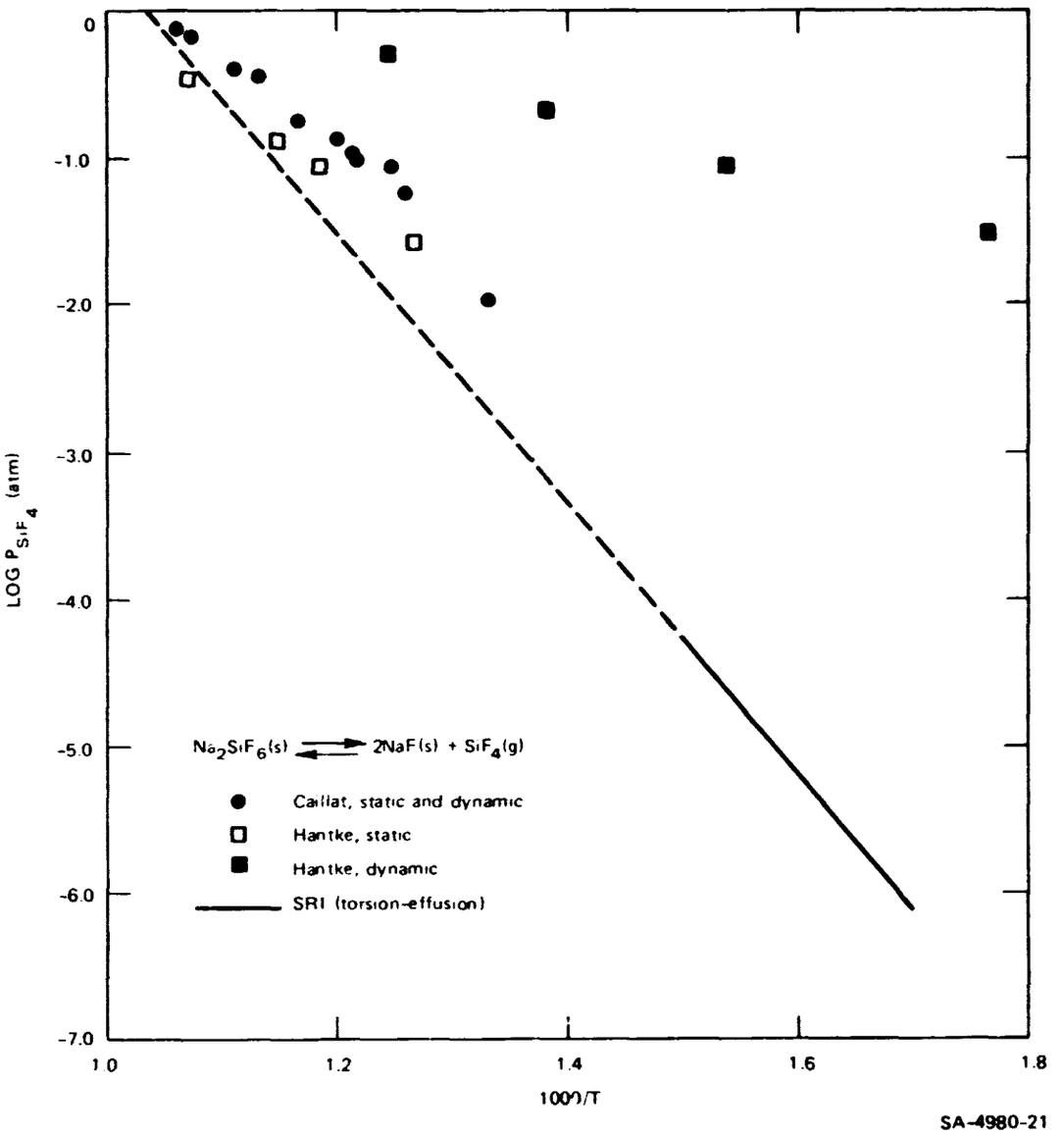

FIGURE 8 EQUILIBRIUM PRESSURE OF SiF_4 VERSUS TEMPERATURE

According to the presently obtained extrapolated relation for the pressure of SiF_4 in Equation 3, an equilibrium pressure of 1 atm of SiF_4 should be reached at 734°C. For a dynamic system, Yaws et al[12] have shown that Na_2SiF_6 dissociates rapidly and completely at 600°C, the highest temperature studied. Further studies at SRI will concentrate on the temperature region 350-700°C.

FUTURE WORK

During the next quarter, we intend to study the leaching process in further detail in order to establish the most suitable conditions for separating silicon from the reaction products NaF and Na_2SiF_6. We also plan to examine candidate methods for the preparation of SiF_4 gas from commercially obtained H_2SiF_6 (ag). Our objective will be to select the most suitable process for obtaining pure SiF_4 gas from H_2SiF_6 economically.

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